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Title: Electrorefining Low Yield Possible Root Causes

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Electrorefining Low Yield

Possible Root Causes

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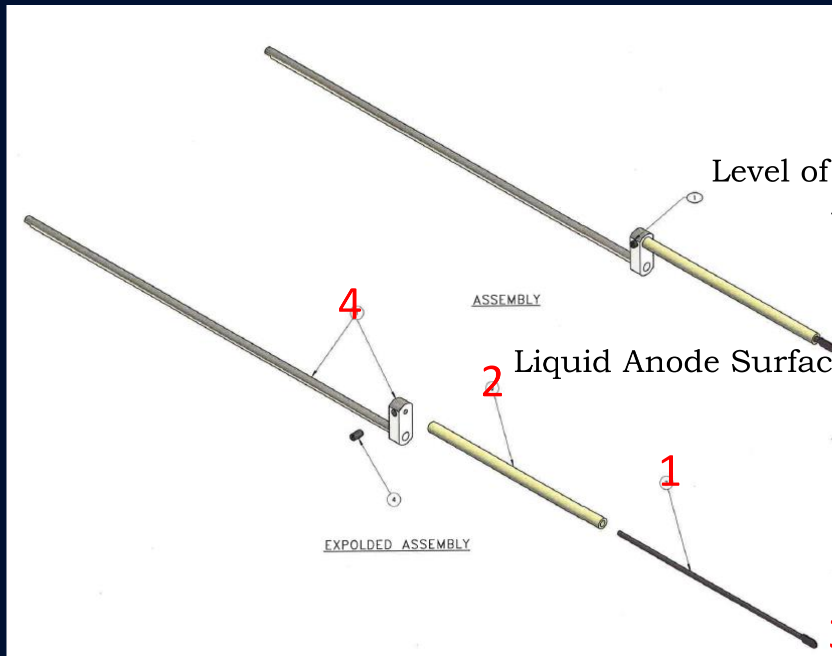
Parts Description

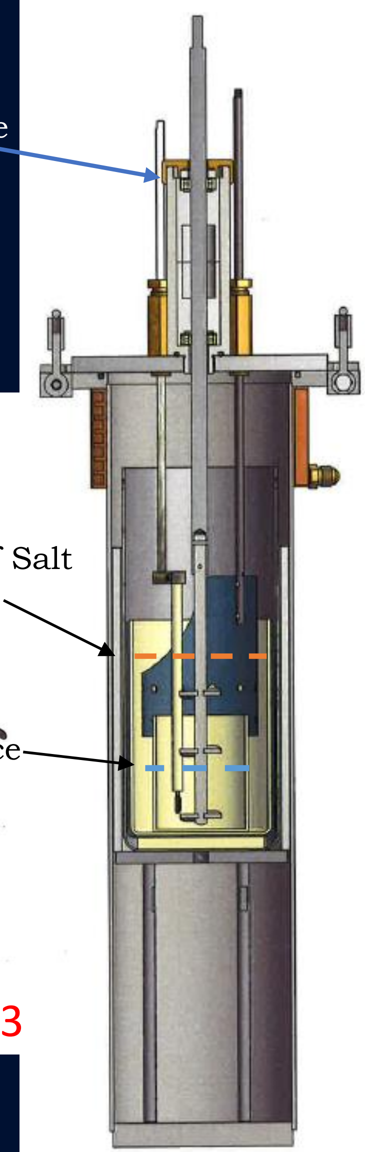
(1) **Tungsten**: electrically conductive, will not oxidize significantly under applied loads (~20A, 2V), refractory metal resistant to Pu corrosion.

(2) **Magnesia Sleeve**: electrically insulates and protects electrode from electrolyte, same material as crucible, resistant to Pu/molten salt corrosion.

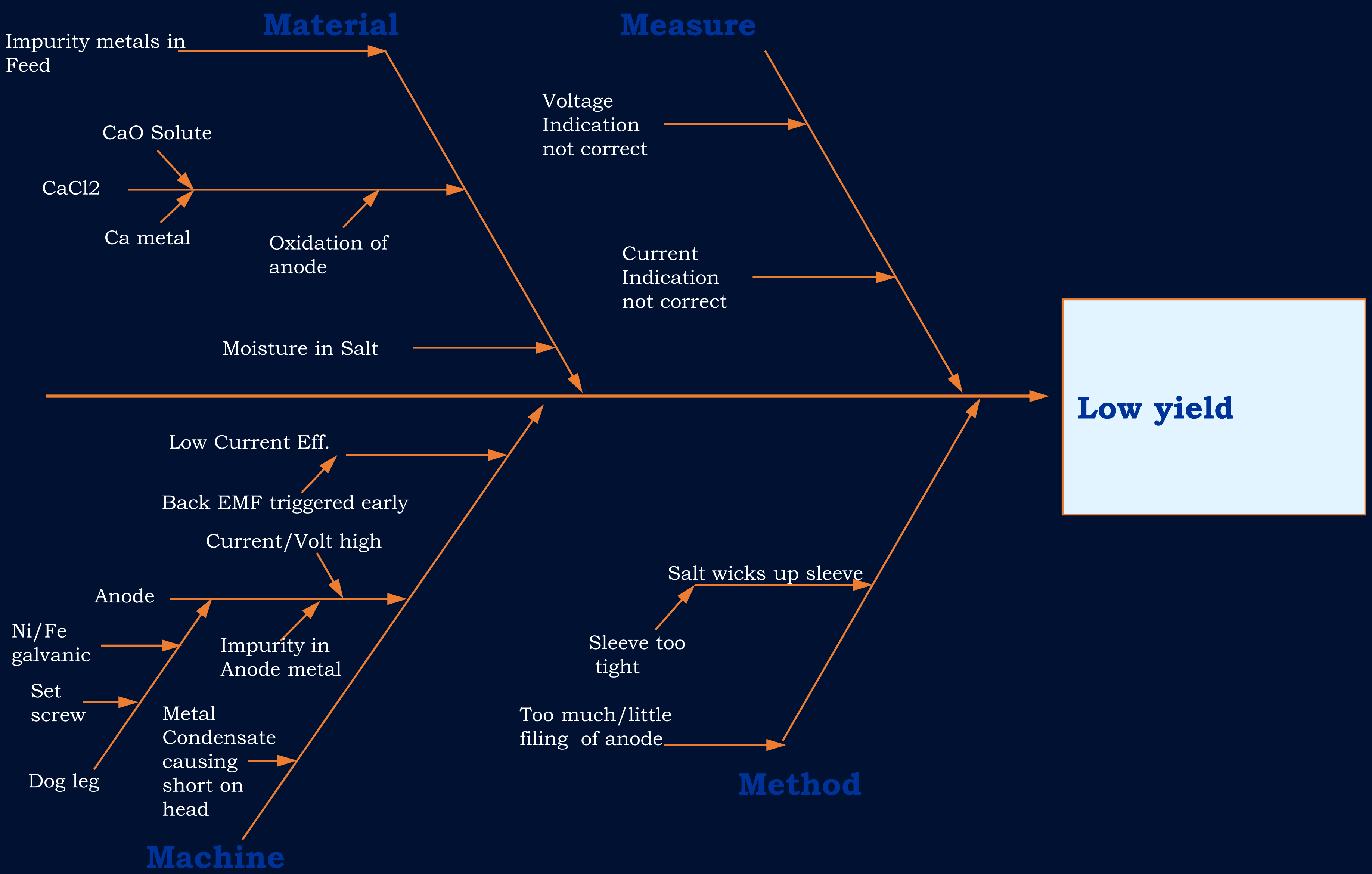
(3) **Paddle Tip**: electrical connection to liquid anode pool, prevents sleeve from falling off.

(4) **Dog Leg**: holds tungsten rod, sits inside furnace, passes current to tungsten, enables positioning rod within crucible cup.

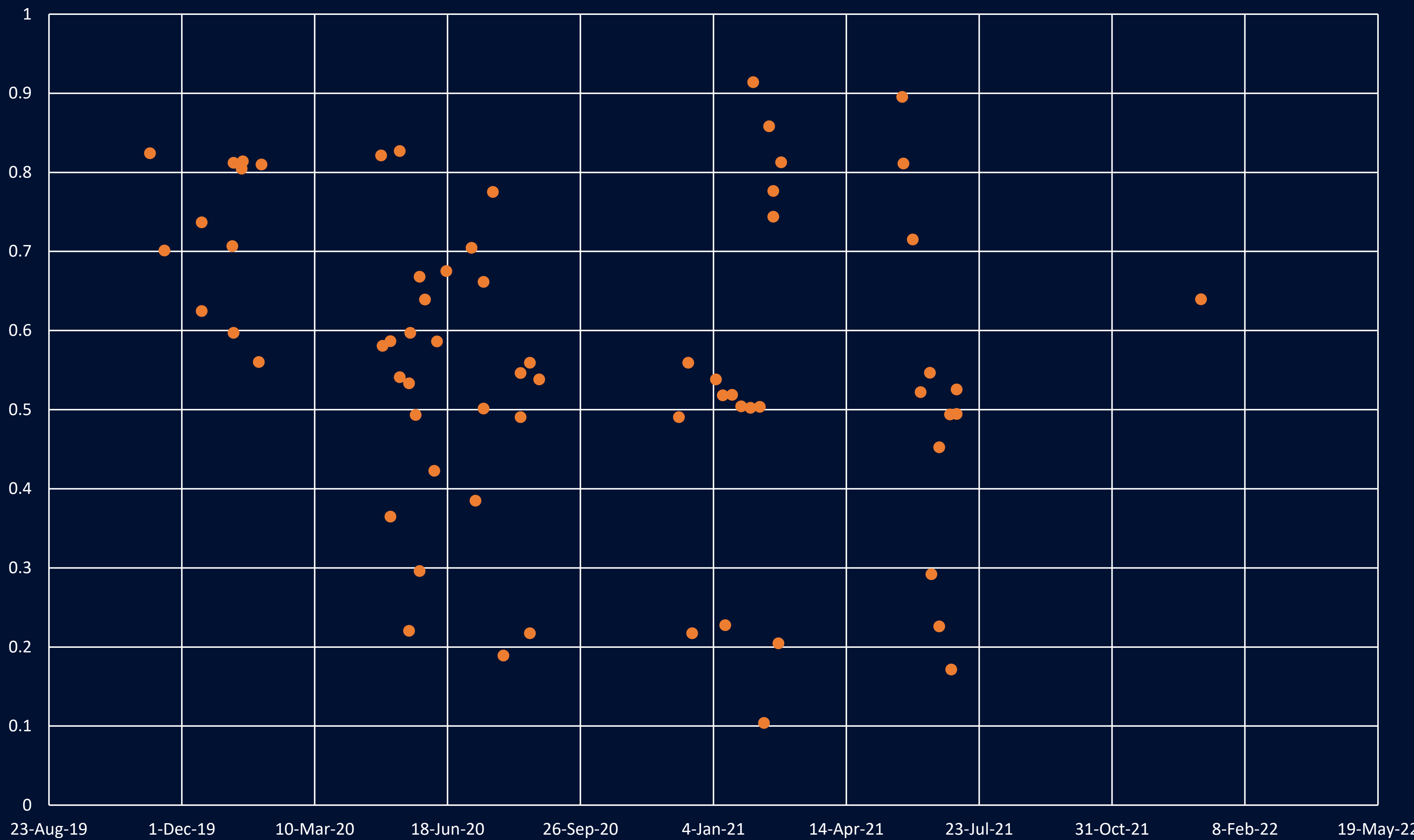




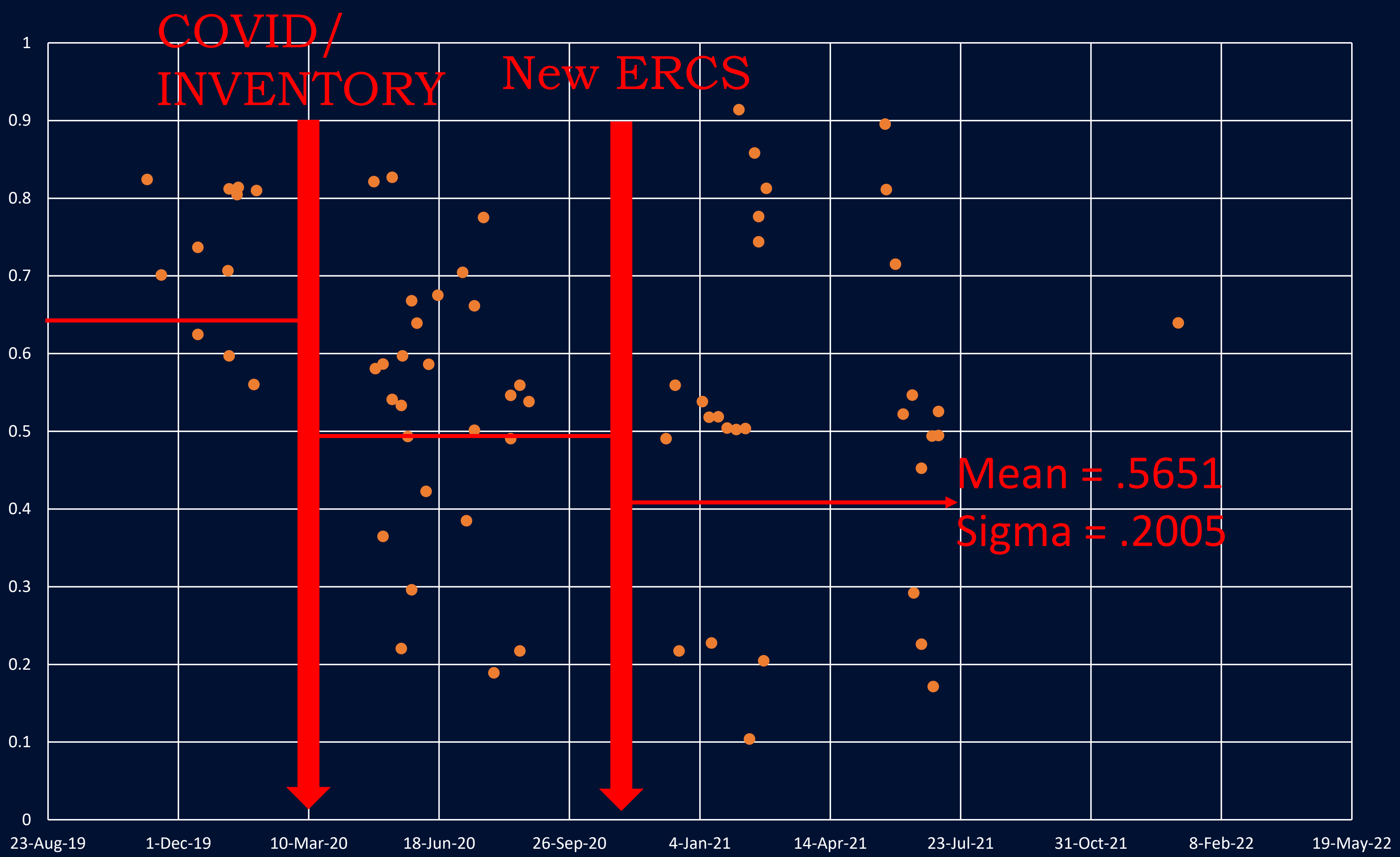
Solving the low yield problem



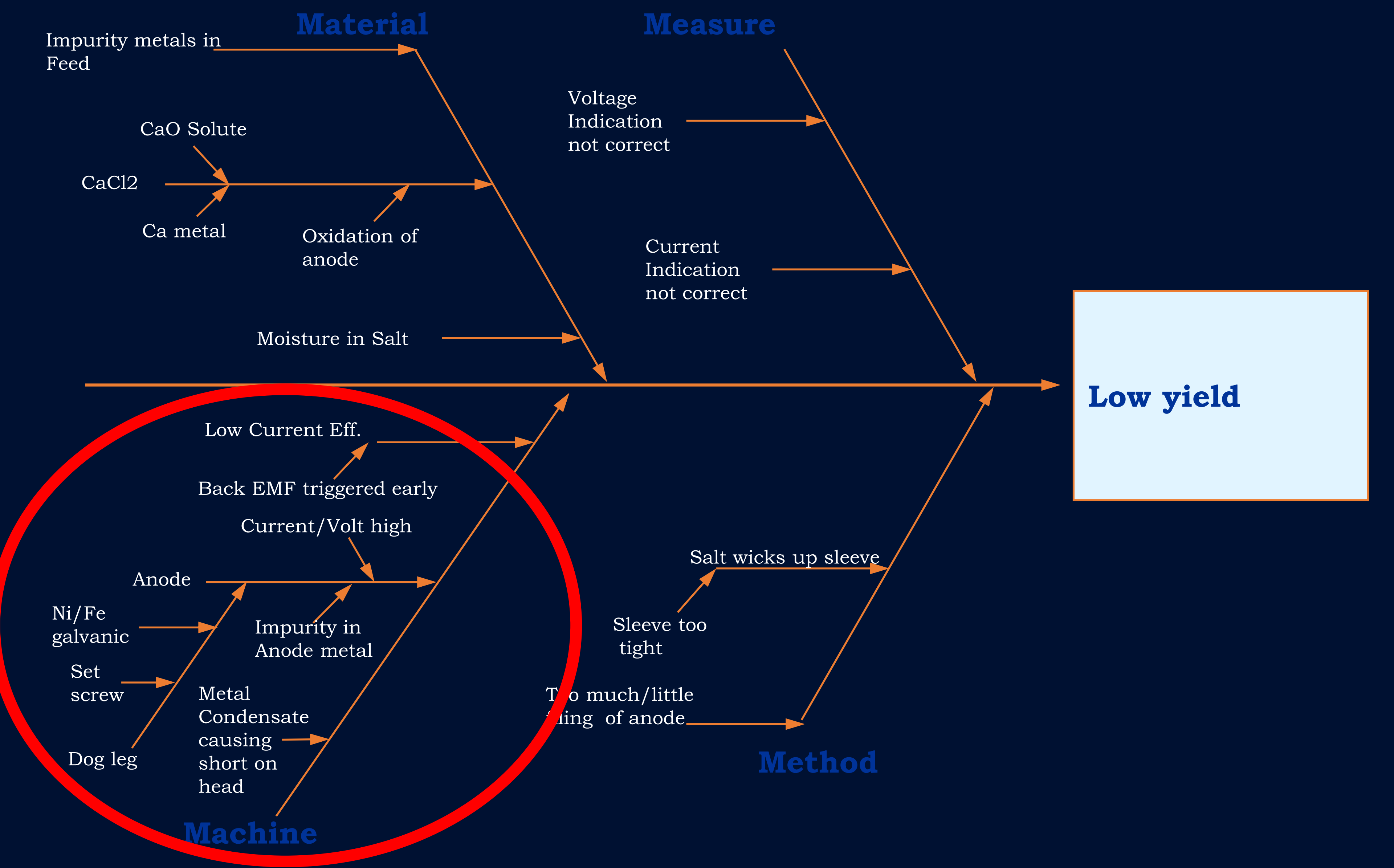
Successful Run Yields FY 20 & 21



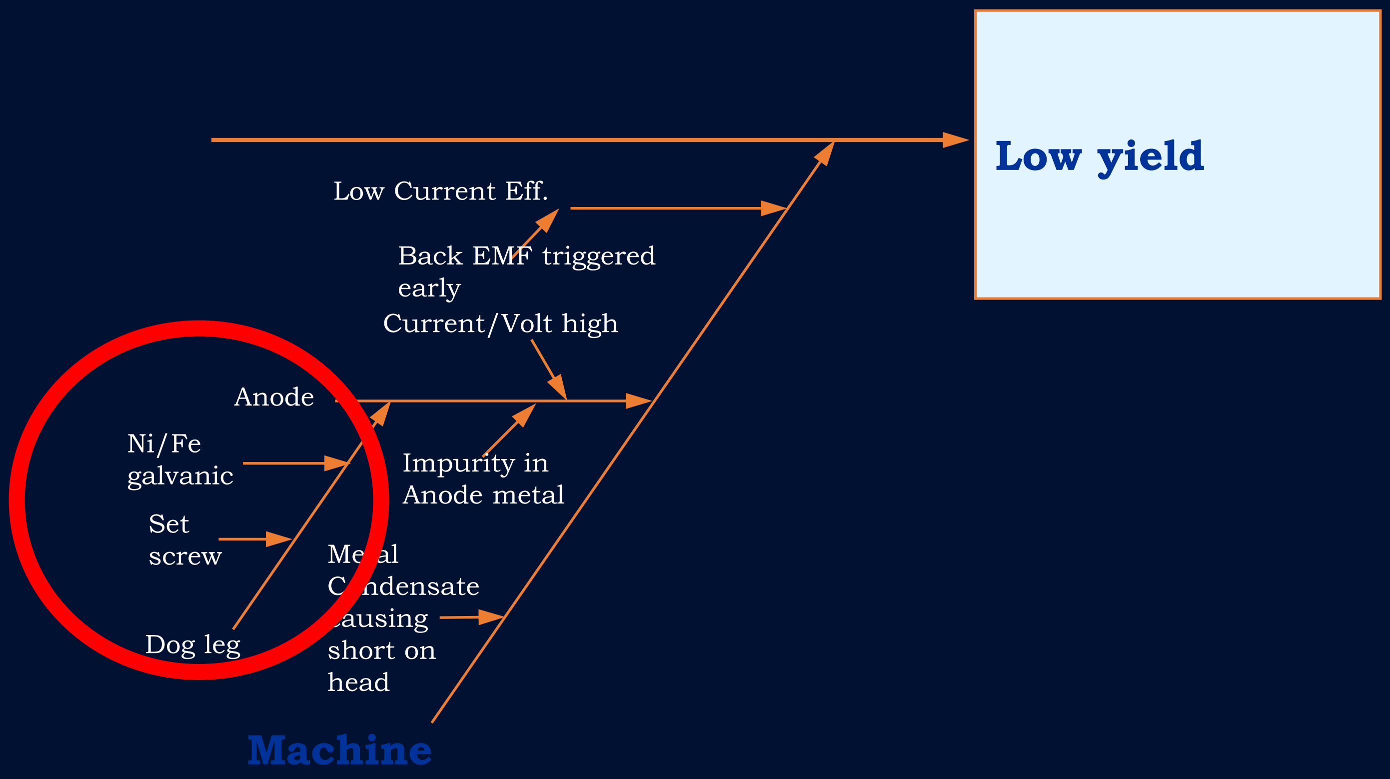
Successful Run Yields FY 20 & 21



Solving the low yield problem



Solving the low yield problem



Problem #1: Paddle Falling Off

-Occurring in the majority of runs in the past 5 months.

-Electrode loses contact with the liquid anode pool, passing current directly into electrolyte, prematurely ending run.

-Sometimes, the ceramic sleeve slides off of rod when raising equipment.

Hypothesis #1

-Tungsten extraordinarily brittle.

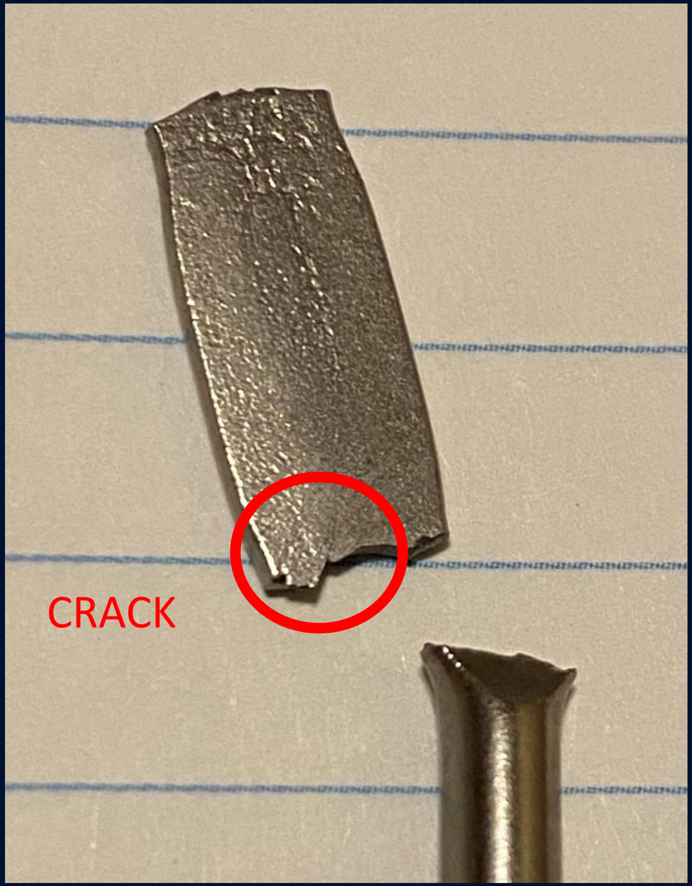
-Very difficult to fabricate with.

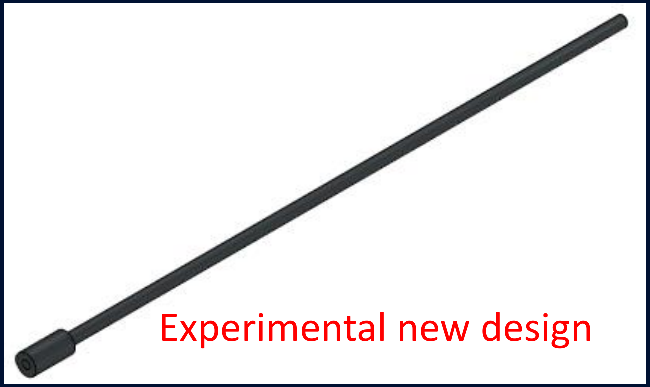
-If not controlled very carefully, material properties negatively impacted (e.g., ductile to brittle transition temperature increases, grain re-crystallization)

OUR FABRICATION WAS NOT CONTROLLED WELL!

-heated with acetylene torch until “red hot”, leading to grain re-crystallization and brittleness.

-press rate for paddle not controlled, resulting in crack defects.





Hypothesis #2

-The anode was lowered into the melt too quickly!

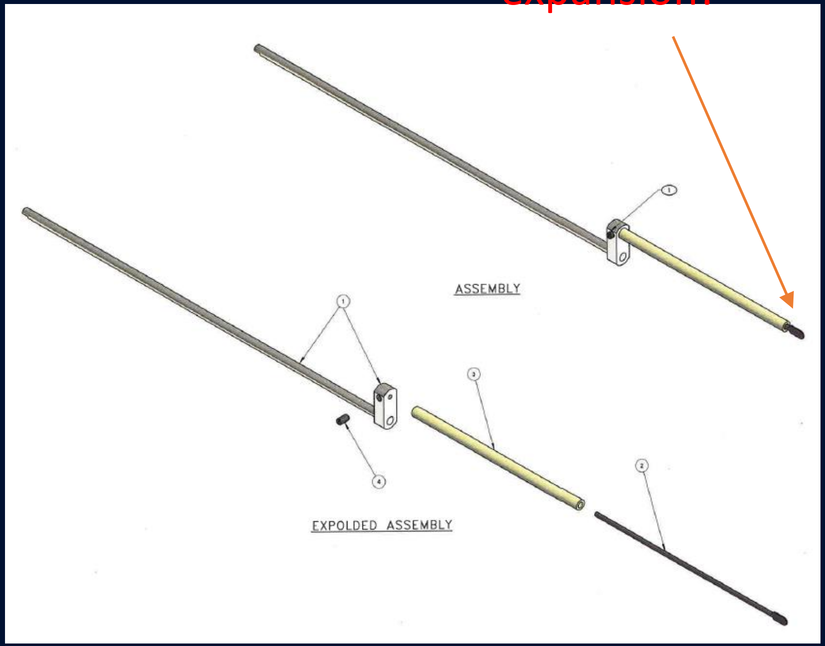
-Thermal gradient induced stresses that fractured the sleeve.

-Thermal expansion coefficient of sleeve is higher than the tungsten. A gap was not left for the sleeve to expand, thus applying stress to sleeve when heated.

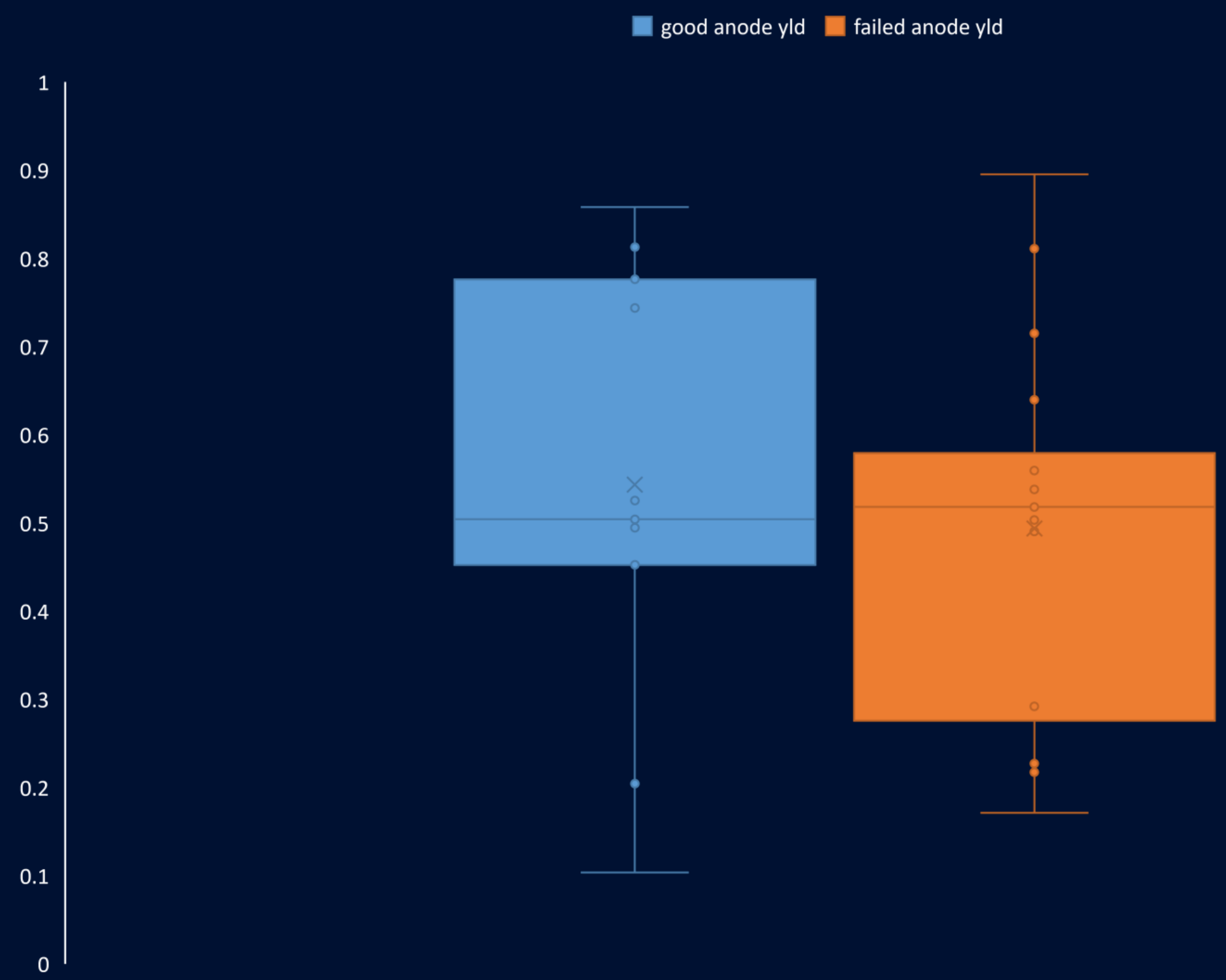
----

1. Change procedure to reduce insertion rate to 0.5 inches per minute (per RFP-1876), and specify 0.125-0.25" gap when assembling anode.
2. Investigating a change from extruded magnesia, which has lower density, to different forming technique.

No gap for expansion!



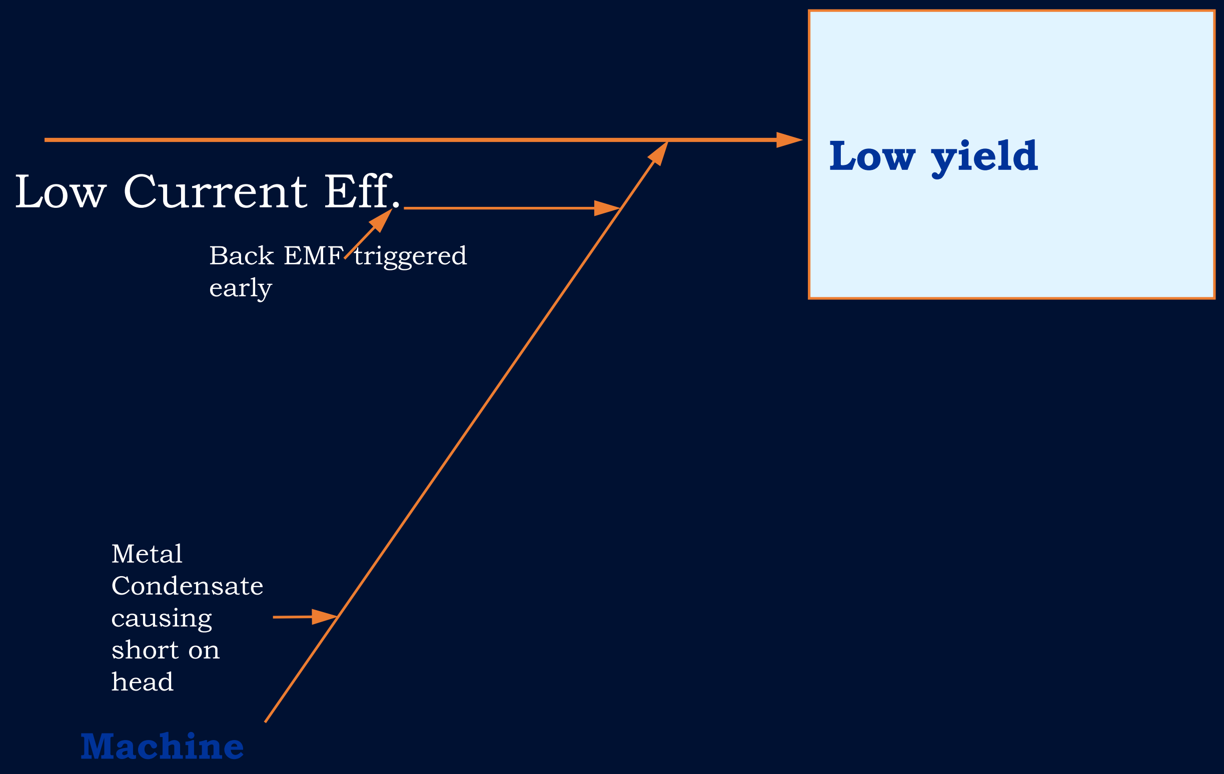
2021 yield failed vs good anodes



T-Tests

t-Test: Two-Sample Assuming Unequal Variances		
	good anode yld	bad anode yld
Mean	0.543413164	0.493556767
Variance	0.058828388	0.041601097
Observations	11	18
Hypothesized Mean Difference	0	
df	18	
t Stat	0.569677385	
P(T<=t) one-tail	0.287968999	
t Critical one-tail	1.734063607	
P(T<=t) two-tail	0.575937999	
t Critical two-tail	2.10092204	
t-Test: Two-Sample Assuming Equal Variances		
	good anode yld	bad anode yld
Mean	0.543413164	0.493556767
Variance	0.058828388	0.041601097
Observations	11	18
Pooled Variance	0.047981575	
Hypothesized Mean Difference	0	
df	27	
t Stat	0.594726428	
P(T<=t) one-tail	0.27848898	
t Critical one-tail	1.703288446	
P(T<=t) two-tail	0.556977959	
t Critical two-tail	2.051830516	

Solving the low yield problem

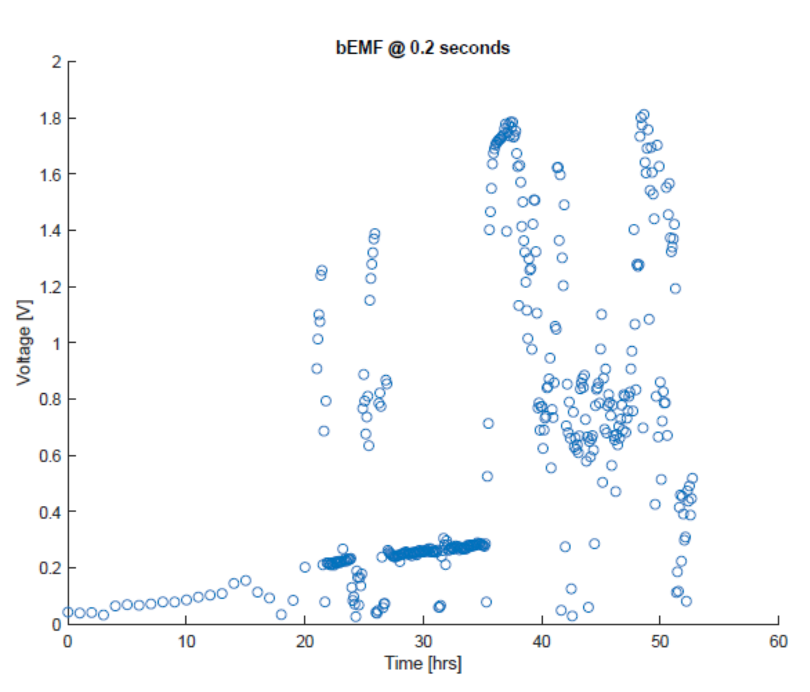


Machine

Problem	Test
• Metal Condensate causing short on head	• Unkown
• Back EMF triggered early?	• Raise back EMF threshold, get chemistry
• Filing Anode too much	• New experimental anode

Problem #3: Premature bEMF Ramp Up

-According to ERCS data, bEMF ramps up earlier in run than historical successful run, resulting in early bEMF termination of the run.



Run from this year

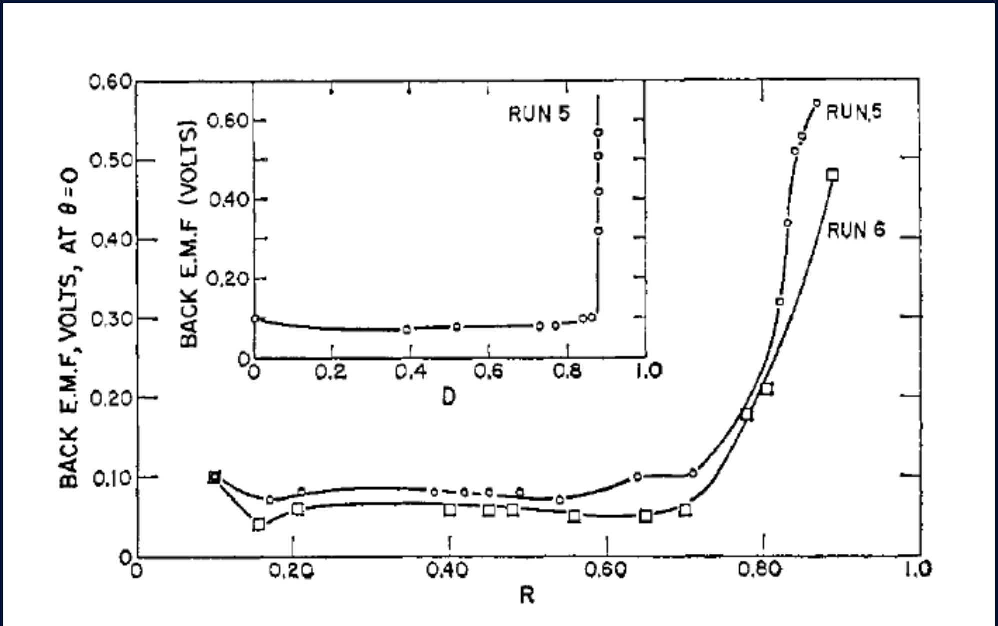
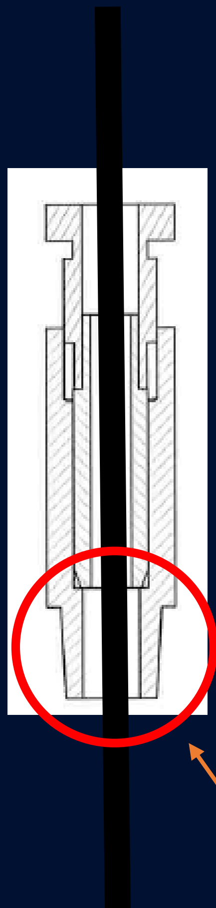


Figure 7. Back-e.m.f. at $\theta = 0$ vs. R for two typical Pu-Fe electrorefining runs

“Textbook” run from 1960s

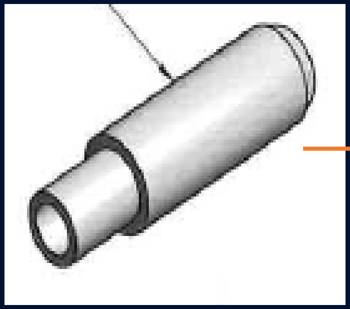
Hypothesis #3



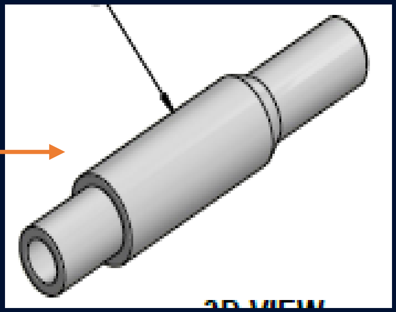
-Teflon piece that insulates electrode and holds in place is not long enough.

-As a run progresses, volatiles deposit on the bottom of the cell head where it is cooler.

-Buildup in the seal gland electrically shorts the anode to the cell head.



Re-design



Build-up and short location

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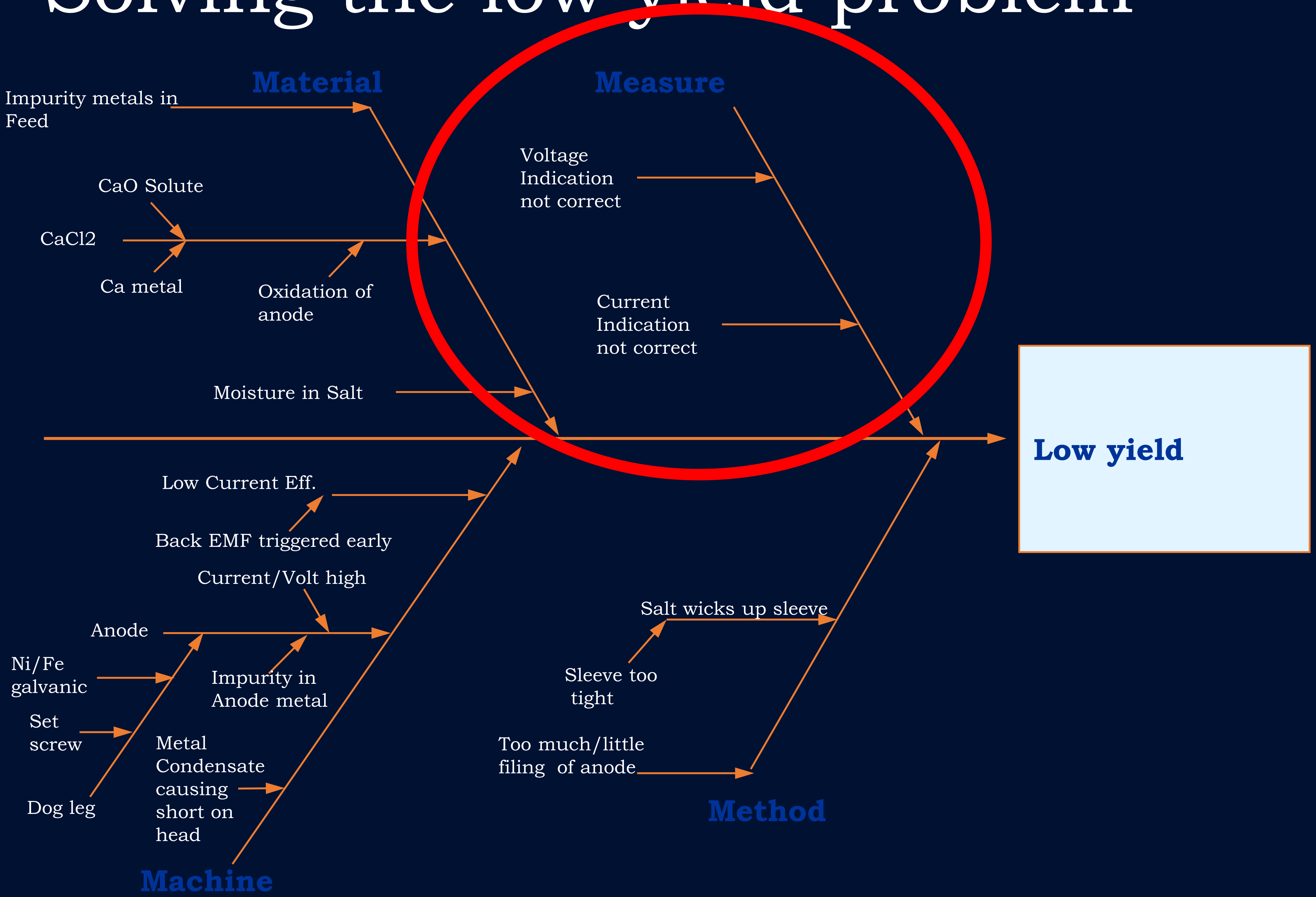

Measure

Problem

- Voltage Indication incorrect
- Current indication incorrect

Test

- Into multi-meter with alligator clips
- Use a current clamp on top of box

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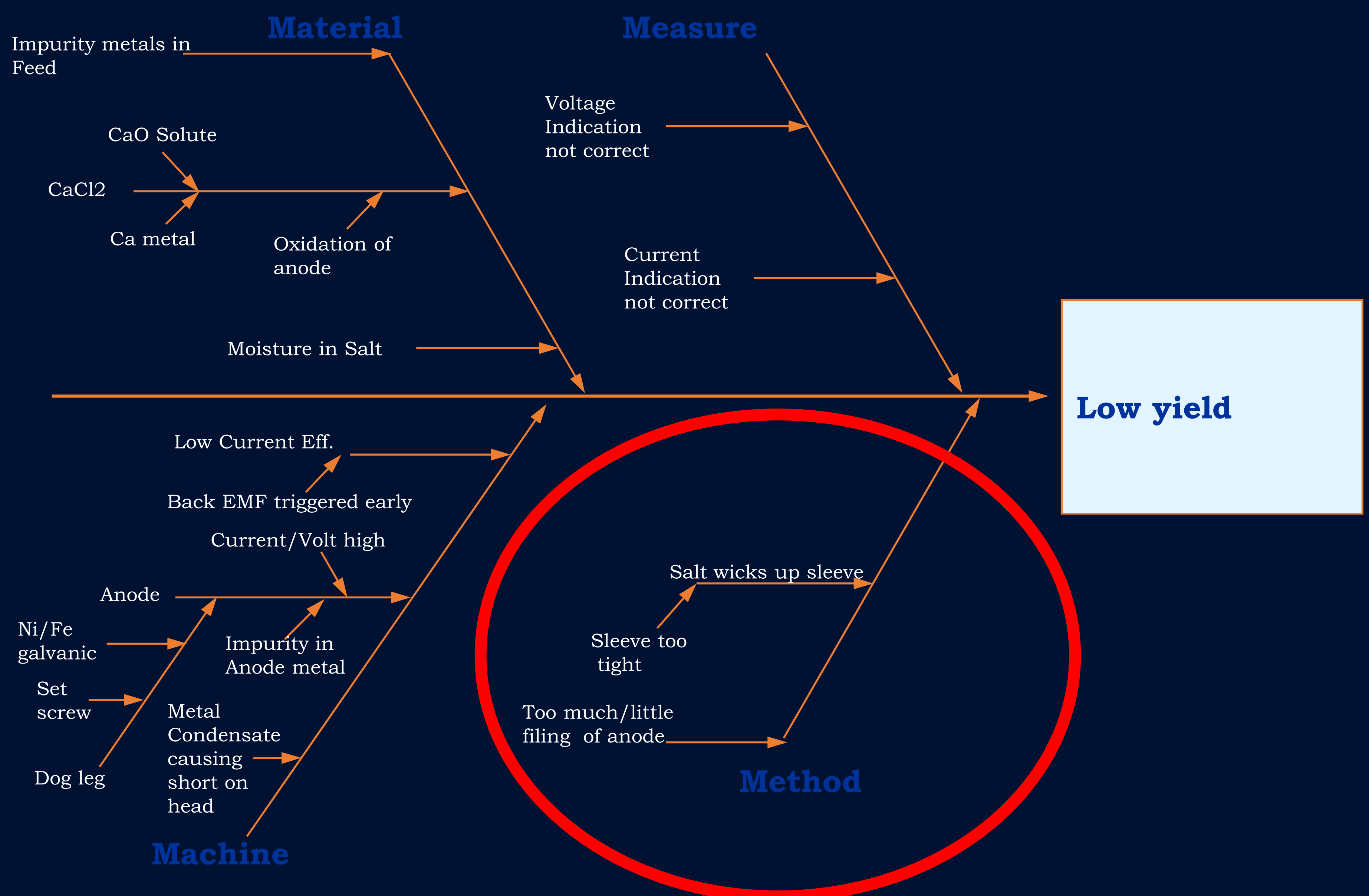


Method

Problem

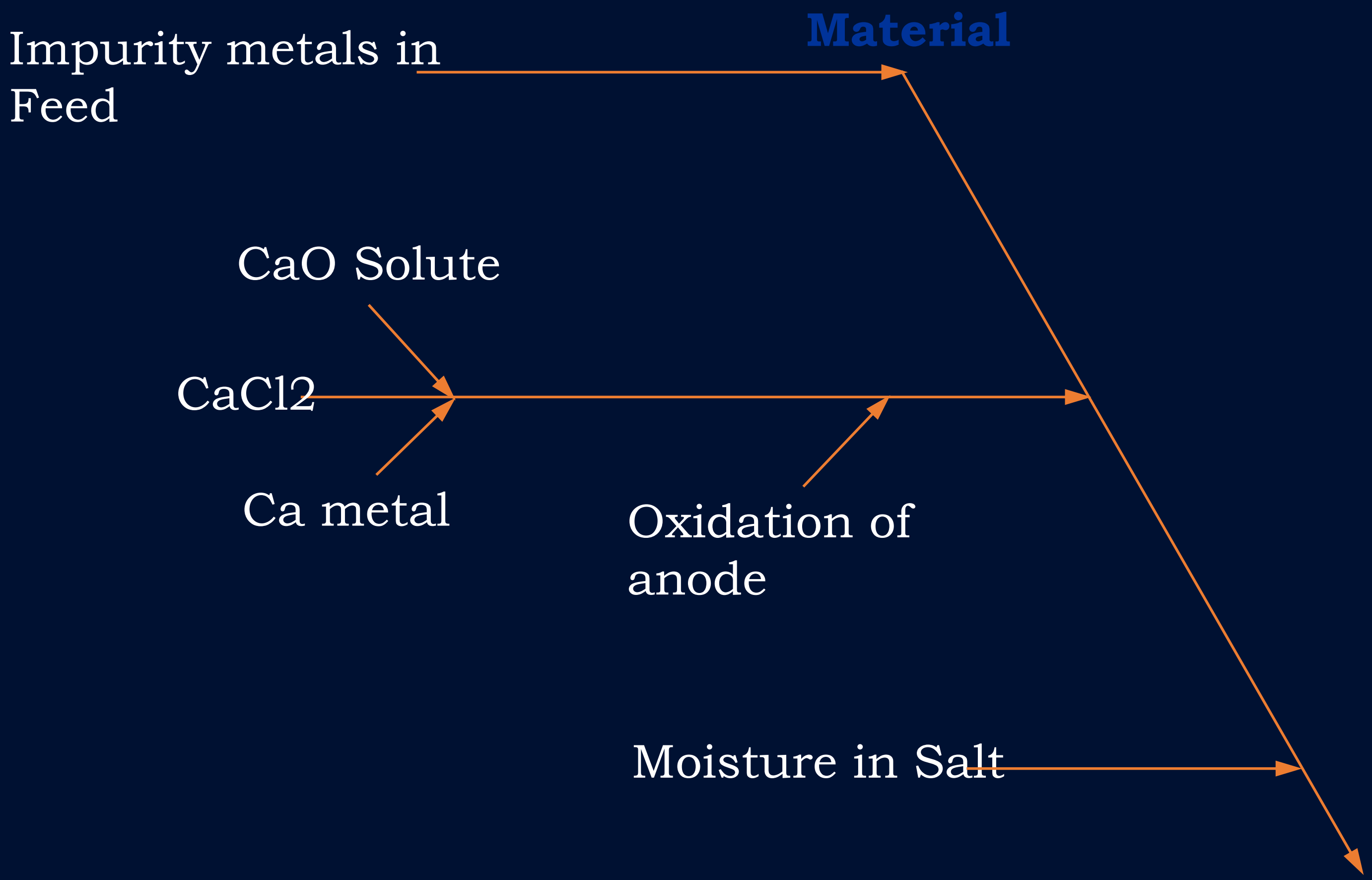
- Sleeve is overtightened and salt wicks up sleeve and starts making contact with dog leg.

Test

- Have a slot gauge to ensure spacing

Solving the low yield problem

Solving the low yield problem ‘



Material

Problem

- All issues related to CaCl2
- Impurity in feed
- Moisture in salt

Test

- temporarily stop using CaCl2
- Regression models chemistry data
- Use new MgCl Salt with eutectic